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Method Name: **Determination of Chlorate and Perchlorate in Baby Foods, Infant/Adult Formulas and Their Ingredients**

Approved by: Stakeholder Panel for Infant Formula and Adult Nutritionals
Final version date:
Effective date:

Intended Use: Surveillance and Monitoring by Trained Technicians

1. **Applicability:**
   Determination of chlorate and perchlorate in baby foods, infant/adult formulas and their ingredients (see Table 3).

2. **Analytical Technique:**
   Any analytical technique that meets the following method performance requirements is acceptable.

3. **Definitions:**
   Accuracy[1]
   The closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value.

   Limit of Quantitation (LOQ)
   The minimum concentration or mass of analyte in a given matrix that can be reported as a quantitative result.

   Repeatability
   Variation arising when all efforts are made to keep conditions constant by using the same instrument and operator and repeating during a short time period. Expressed as the repeatability standard deviation (SD_r); or % repeatability relative standard deviation ($\%RSD_r$).

   Reproducibility
   The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility relative standard deviation (SD_R); or % reproducibility relative standard deviation ($\%\ RSD_R$).

   Adult/Pediatric Formula
   Nutritionally complete, specially formulated food, consumed in liquid form, which may constitute the sole source of nourishment, made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

[1] Corresponds to the VIM definition for “trueness”.

Baby food
Food intended for use by infants when they are weaned and by young children as a supplement to their diet and/or for their progressive adaptation to ordinary food. These include cereal-based products sold as dry, to be reconstituted before consumption or wet meals ready to eat, sweet or savory.[2]

Infant formula
Breast-milk substitute specially manufactured to satisfy, by itself, the nutritional requirements of infants during the first months of life up to the introduction of appropriate complementary feeding[3], made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

Ingredients
Constituents of the above products used in their formulations (see Table 3).

Chlorate
Chlorate anion ClO_3^- (CAS 14866-68-3)

Perchlorate
Perchlorate anion ClO_4^- (CAS 14797-73-0)

4. Method Performance Requirements:

Table 1. Limit of quantitation (LOQ)

	Chlorate	Perchlorate
Baby foods* and their major ingredients	≤ 0.005 mg/kg	≤ 0.005 mg/kg
Infant/adult formulas*	≤ 0.005 mg/kg	≤ 0.005 mg/kg
Infant/adult formula ingredients – major (> 10% in finished powdered product)**	≤ 0.01 mg/kg	≤ 0.01 mg/kg
Infant/adult formula ingredients – minor (≤ 10% in finished powdered product)**	≤ 0.1 mg/kg	≤ 0.1 mg/kg
Infant/adult formula ingredients – liquid milk	≤ 0.005 mg/kg	≤ 0.005 mg/kg

*Concentrations apply to as consumed products. For infant formulas, these include:
a) "ready-to-feed" liquids "as is";
b) re-constituted powders (based on the product instructions or using a generic reconstitution of 25 g into 200 g of water).

**The ingredient content of 10% in a finished powdered infant/adult formula product corresponds to about 1.1% content in a ready-to-feed or reconstituted (as consumed) product, using the generic reconstitution factor of 25 g into 200 g of water. Examples of major and minor ingredients are whole milk powder and premixes, respectively.

Table 2. Recovery, repeatability and reproducibility parameters

Recovery	80-120%
Repeatability (RSD_r)	≤ 20%
Reproducibility (RSD_R)	≤ 25%

See also Section 7: Validation Guidance.

[2] European Commission Food Labeling Guidelines. https://ec.europa.eu/food/safety/labelling_nutrition/special_groups_food/children_en

[3] Codex Standard 72 – 1981.

5. System suitability tests and/or analytical quality control:

Suitable methods will include blank check samples and appropriate check standards. Method (procedural) and solvent blanks should be below 0.3 x LOQ.

6. Reference Material(s):

Source	Product	Chlorate (mg/kg)	Perchlorate (mg/kg)
NIST	SRM 1869 Infant/Adult Nutritional Formula II (milk/whey/soy based) *coming soon*	0.105	--
NIST	RM 8260 Infant Formula (hydrolyzed protein based) *coming soon*	0.281	--
FAPAS	T05140QC Infant Formula	--	0.052
FAPAS	T19298QC Salad Leaf	0.497	0.338
FAPAS	Infant Formula (2021 PT Round 05157)	--	?

Additionally, remainder materials (infant formulas and infant formula ingredients) tested as part of a NIST interlaboratory comparison may be available for method developers.

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements*, 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_f.pdf

7. Validation Guidance:

Validation (including criteria from Section 4: Method Performance Requirements) should be conducted at least at the target LOQ and 10xLOQ levels. The LOQ is determined as the lowest spiking level that meets the recovery and repeatability requirements. Suitable matrix blanks should be selected that do not contain more than 30% of the target LOQ level for each analyte.

For matrices that contain incurred levels of chlorate and where suitable matrix blanks are not available, spiking experiments should be conducted at two concentration levels in the range of 3-10x the analyte level in the evaluated matrix. The LOQ can then be estimated based on extrapolation of signal-to-noise ratio (S/N) obtained for a concentration level present in the evaluated matrix to a concentration level that would correspond to S/N = 10.

Additional guidance can be found in the following resources:

SANTE guidelines on "Analytical quality control and method validation procedures for pesticide residues analysis in food and feed" issued by the European Commission Directorate General for Health and Food Safety (SANTE/12682/2019 or the recent version).

Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www. eoma.aoac.org/app_f.pdf).

8. Maximum Time-to-Result:

No maximum time.

Table 3. Target matrices*

Matrix Category	Matrix Sub-Category	Minimum Representative Matrices
Baby food		• Cereal-based • Fruit/vegetable-based • Meat-based
Baby food ingredients		• Cereal-based • Fruit/vegetable-based • Meat-based
Infant/adult formulas		• Milk-based • Plant-based
Infant/adult formula ingredients	Animal-based milk powdered protein sources	• Whole milk powder • Whey protein concentrate
	Plant-based protein sources	• Soy protein isolate
	Liquid milk	• Bovine • One additional species
	Fat-based	• Oil/fat
	Carbohydrate-based	• Lactose • Maltodextrin • Oligosaccharides (galacto-oligosaccharides or fructo-oligosaccharides)
	Mineral- and vitamin-based (premixes)	• Any

*In order to include the given matrix category in the method applicability, the validation has to be conducted on all representative matrices listed as the minimum requirement for that matrix category. For example, a method has to be validated on lactose, maltodextrin and oligosaccharides (galacto-oligosaccharides or fructo-oligosaccharides) to demonstrate applicability for the carbohydrate-based infant/adult formula ingredient category.